



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/EP97/01880 <b>(22) International Filing Date:</b> 11 April 1997 (11.04.97) <b>(30) Priority Data:</b> 9607600.5                      12 April 1996 (12.04.96)                      GB 9618658.0                      6 September 1996 (06.09.96)                      GB <b>(71) Applicant (for all designated States except US):</b> BRISTOL-MYERS SQUIBB COMPANY [US/US]; 345 Park Avenue, New York, NY 10154 (US). <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> KERSHAW, David [GB/GB]; The Flat, Greyhound Vaults, Market Street, Abergavenny, Gwent NP7 5SD (GB). MAHONEY, Peter, M., J. [GB/GB]; Lletty Meirch Farm, Llanfihangel-Ng-Nhwyntfa, Near Llanfyllin, Powys SY22 5JF (GB). HANMER, Paul [GB/GB]; Brackenwood, Alltarni Road, Buckley, Flintshire CH7 3PD (GB). PRITCHARD, David [GB/GB]; 18 Parc-Y-Fro, Creigau, Cardiff, South Glamorgan CF4 8SA (GB). <b>(74) Agent:</b> MAYS, Julie; Bristol-Myers Company Limited, Patent Dept., Swakeleys House, Milton Road, Ickenham, Uxbridge UB10 8NS (GB).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> COMPOSITE FIBRES, WOUND DRESSINGS INCORPORATING SUCH FIBRES AND A METHOD FOR MAKING SAME		
<b>(57) Abstract</b> <p>An absorbent, composite fibre comprising a matrix of from 10 % to less than 50 % of water insoluble alginate having dispersed therein at least 40 % of another polysaccharide.</p>		

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5        COMPOSITE FIBRES. WOUND DRESSINGS INCORPORATING SUCH  
         FIBRES AND A METHOD FOR MAKING SAME

         The invention relates to composite fibres, particularly  
absorbent composite fibres for use in wound treatment,  
10       wound dressings incorporating such fibres and a method for  
         making same.

         Absorbent fibres for use in wound treatment are well known  
in the art. Examples include cellulose fibres, chemically  
15       modified cellulose fibres, pectin fibres, alginate fibres,  
         chitosan fibres, hyaluronic acid fibres or other  
         polysaccharide fibres or fibres derived from gums. In the  
         treatment of wounds it is desirable to use fibres made from  
         pectin or carboxymethyl cellulose but the known processes  
20       for making such fibres are complex and expensive and the  
         resulting fibres not always viable. For instance it is  
         known to make carboxymethyl cellulose fibres by chemically  
         converting preformed cellulose fibres. It is also known  
         that both pectin fibres and carboxymethyl cellulose fibres  
25       are difficult to spin.

         It has been proposed in GB 2062652 A to make cellulose  
fibres comprising anionically modified polysaccharides by  
adding the polysaccharide to preformed viscose. Similarly  
30       in US 4063558 there is described a method for making fibres  
         of cellulose with alkali metal salts of alginic acid  
         uniformly dispersed therein by adding a solution of sodium  
         alginate to preformed viscose.

- 5 In WO 96/10106 there are described fibres which preferably comprise from 70 to 95% by weight of an alginate co-spun with from 5 to 30% by weight of at least one water soluble organic polymeric species (other than alginate).
- 10 We have now found that it is possible to make a composite, absorbent fibre comprising a matrix of water insoluble alginate having another polysaccharide dispersed therein where the fibre comprises less than 50% by weight of the alginate, the fibre mitigating the disadvantages of the
- 15 prior art fibres.

Accordingly the invention provides an absorbent, composite fibre comprising a matrix of at least 10% and less than 50% by weight of water insoluble alginate having dispersed

20 therein at least 40% of another polysaccharide. Unless otherwise stated all percentages herein are by weight based on the weight of the fibre.

Whilst not wishing to be bound by theory it is believed

25 that the water insoluble alginate effectively provides, as a matrix, a molecular backbone to the fibre that enables the other polysaccharides to be incorporated and results in a fibre that may be spun and otherwise processed. The use of water insoluble alginate for this purpose enables the

30 fibres to be made without the need for complex and expensive processing and without the need to use preformed cellulose as a starting material. Preferred polysaccharides for addition to the alginate backbone are carboxymethyl cellulose and/or pectin.

5 Since it is believed that the fibres of the invention rely on the water insoluble alginate to provide integrity to the fibres it is truly surprising that it is possible to make viable fibres processable into products that comprise a minor proportion of insoluble alginate. One of the  
10 advantages of such fibres is that they may comprise a major proportion of polysaccharide other than alginate which generally makes them more absorbent than fibres which have insoluble alginates in a major proportion.

15 Preferably the fibres comprise, in addition to insoluble alginate, from 40% to 90% of another polysaccharide, more preferably from 60% to 85% and most preferably from 70% to 80% of another polysaccharide which is most preferably carboxymethyl cellulose or pectin or a mixture thereof.  
20 Polysaccharides suitable for use in fibres according the invention include carboxymethyl cellulose, carboxyethyl cellulose, other derivatives of cellulose, cellulose, pectin, hyaluronic acid and chitosan. Preferably the insoluble alginate is calcium alginate.

25 Preferably the fibres comprise from 10% to less than 50% by weight of the fibre of insoluble alginate, more preferably 10% to 49%, more preferably 15% to 40% and most preferably from 20% to 30% by weight of the fibre of water insoluble  
30 alginate. Preferably the insoluble alginate is calcium alginate.

A particularly preferred embodiment of the absorbent, composite fibres of the invention comprise a matrix of 20%

5 to 30% of calcium alginate based on the weight of the fibre having dispersed therein from 55% to 60% of carboxymethyl cellulose and 15% to 20% of pectin based on the weight of the fibre.

10

Accordingly in another aspect the invention provides a method for making a composite, absorbent fibre comprising the following steps:

- 15 (i) adding sodium alginate and another polysaccharide to water to form a dope;
- (ii) forcing the dope through a spinneret to form fibres;
- (iii) treating the resulting fibres with a source of calcium ions to convert the alginate to calcium alginate and cross-link the alginate to the other polysaccharide;
- 20 (iv) drying the fibres.

The process enables composite fibres to be prepared which comprise large quantities of other polysaccharides and in particular, carboxymethyl cellulose, pectin or both. The fibres so produced are capable of being spun or otherwise mechanically processed. In addition the resulting fibres have a high tensile strength compared to alginate, carboxymethyl cellulose or pectin alone.

30 As used herein the term fibre means both relatively short, discrete, randomly oriented material (sometimes known as staple fibre) and yarns made therefrom (sometimes known as staple yarn) and relatively long, structured, continuous filament yarn. The fibres may have a staple length of 5mm

5 to 70 mm, more usually 20mm to 50 mm, favourably 25mm to 40 mm.

The fibres prepared according to the above described process may be dried using conventional methods, for  
10 example, using acetone or hot air drying.

Alginates are produced by a variety of micro-organisms and marine algae which are the normal commercial source. The alginates being natural materials show considerable variety  
15 but are characterised in being block copolymers, the individual monosaccharide units being arranged into groups as blocks of mannuronic (M) and guluronic (G) residues. In addition to the repeating blocks each polymer chain can contain a proportion of alternating M and G monosaccharide  
20 units. The alginate may be obtained from any convenient source, for example L. Hyperborea or Ascophyllum Nodosum or those described in EP-A-0721355 to Bristol-Myers Squibb Company which are particularly preferred.

25 In a further embodiment of the invention the absorbent, composite fibres are employed in the manufacture of wound dressings. Accordingly the invention provides a wound dressing comprising an absorbent, composite fibre  
30 comprising a matrix of between at least 10% and less than 50% of water insoluble alginate having dispersed therein at least 40% of another polysaccharide.

The wound dressings may be in the form of swabs, wound

5 pads, wadding ribbons, sponges, nets and bandages and may  
be used as a primary or secondary dressing especially in  
the treatment of leg ulcers. The wound dressings according  
to the invention may benefit from an improved integrity  
over that of dressings made from alginate, pectin or  
10 carboxymethyl cellulose alone.

According to a further aspect of the invention there is  
provided a wound dressing comprising a mixture of discrete  
textile fibres and discrete absorbent, composite fibres  
15 said absorbent, composite fibres comprising a matrix of at  
least 10% of water insoluble alginate having dispersed  
therein at least 40% of another polysaccharide. Such a  
dressing may have the advantage that it is non-adherent to  
wound tissue while being absorbent and relatively  
20 inexpensive and the added advantage that it may be retained  
on the wound for longer than conventional cotton gauze.  
The absorbent, composite fibres according to the invention  
incorporated in the wound dressing become moist and  
slippery or gelatinous upon the uptake of wound exudate.  
25 This reduces the tendency for the textile fibres to adhere  
to the wound.

In general textile fibres absorb liquids by capillary  
action and are not hygroscopic. This means that their  
30 absorbencies as measured by the free swell absorbancy test  
are low such as less than 1 gram of liquid per gram of  
fibre. Suitable textile fibres can be natural or synthetic  
depending on the end use of the dressing and method of  
manufacture. Suitable textile fibres are for instance



5 described in PCT/GB95/00114. For example where the dressing is made from a non-woven mixture of discrete fibres the textile fibre is preferably one that can be fused at relatively low temperatures, for example polypropylene. The entire dressing can be heat fused to  
10 give a dressing with sufficient tensile strength that it may be removed intact from the wound even though saturated with exudate. This avoids the need for the painstaking removal from the wound of parts of a dressing that has lost its integrity on exposure to exudate.

15

Where the dressing is made from a woven mixture of discrete fibres the textile yarn can be polyester, polypropylene or polyamide or any other suitable support yarn. We have surprisingly found that it is possible to knit fibres of  
20 the invention in a knitting process where it is preferable for the textile yarn to form the pillar or chain stitches of the knit (the warp) and the composite fibre to form or be included in the the in-laid yarn of the knit. A particularly suitable knit of this type is a Raschel knit  
25 as described in Textile Science by Kathryn Hatch, West Publishing Company 1993. If composite fibres are included in both the pillar and the in-laid yarn then the pillar yarn tends to break and shed fibres. If composite fibres are included only in the in-laid yarn then this tendency is  
30 overcome and quite high machine knitting speeds can be achieved and a dressing of better integrity is produced.

According to a further aspect the invention provides a wound dressing as claimed in claims 11 to 14 wherein the

5 dressing is in the form of a warp knitted fabric comprising pillar yarn and in-laid yarn, the pillar yarn being substantially free of composite fibres.

Various optional ingredients can be included in the final  
10 composition of the fibres such as preservatives and small amounts of pharmacologically active ingredients. For example an antibiotic or antimicrobial agent such as metronidazole, silver sulphadiazine, neomycin or penicillin and antiseptic agent such as povidone iodine, iodine and  
15 antiinflammatory agent such as hydrocortisone or triamcinolone acteonide or a skin protective agent such as a zinc oxide can be included.

The invention is illustrated by the following examples:-

20

Example 1

Fibres according to the invention in the form of a continuous yarn were prepared as follows:

800ml of a 6% w/w dope was made by adding 28g of  
25 carboxymethyl cellulose (ex Hercules), 12g of alginate (ex Kelco), and 8g of pectin (ex Aldrich Ltd) to 752g of deionised water. The mixture was stirred with a high speed mixer until the ingredients had dissolved and the dope allowed to stand overnight to degas.

30

The degassed dope was then poured into a pressure vessel connected to the dope pumping/filtering system of a spinning rig. The pressure vessel was pressurised to 2 to 3 atmospheres with compressed air and the pump, 60µm pore

5 size filter and associated pipe work purged to remove any  
air bubbles. A 400 jet spinneret, previously  
ultrasonically cleaned for 20 minutes, was connected to the  
rig and the rig started. On exit from the spinneret, the  
dope was fed into a spin bath of 2m length containing 30L  
10 of 0.4 mol/dm<sup>3</sup> of calcium chloride. The fibres so produced  
were threaded over three sets of rollers of a first godet  
and then over the rollers of a second godet. Propan-2-ol  
was dripped onto the fibres at a rate sufficient to wet the  
resulting yarn. The yarn was then passed into two baths,  
15 the first contained 4L of propan-2-ol and the second  
contained 2.5L of propan-2-ol. The propan-2-ol was  
maintained at a concentration to dry the yarn. The yarn  
was then passed through a set of pinch rollers that serve  
to apply tension to yarn. The yarn was then wound onto  
20 cones. The dope was supplied to the system at 6ml per  
minute and a speed differential maintained between the  
first and second godets resulted in a stretch of 60%. The  
resulting yarn had a denier of 300 to 400.

25 Example 2

A knitted wound dressing according to the invention was  
prepared comprising the yarn of example 1 and a crimped  
polyester yarn. The dressing was knitted on a crochet  
knitting machine (Model STP7 ex KOHLER) each needle of  
30 which creates a chain of interlocked loops (pillar or clain  
stitches). These form the warp threads of the dressing.  
The warp threads are held together by weft threads. 45 warp  
threads of stiches were knitted from 150 denier crimped  
polyester yarn. These were held together by 44 threads of

5 the yarn of example 1 (in-laid yarn) to form a dressing.

#### Example 3

A non-woven wound dressing according to the invention was made by mixing 200g of the absorbent, composite fibres of example 1 cut into 5cm lengths and 200g of polypropylene staple fibre cut to 5cm lengths in a rag roller to randomise the fibre. The mixture of fibres was then carded and cross-lapped into a web of 100gsm basis weight. The web was then passed through heated callender rollers at 65°C and pressure. The resultant product was slit into discrete dressings 10cm by 10cm square.

#### Example 4

A staple yarn was made from the composite fibres of the invention in the following way. A mixture of the composite fibre (20kg) and polypropylene (20kg) (all 3 denier) was cut to a staple length of 40mm and converted into a lap of approximately 100gsm on a conventional short staple scutching line - a Truteschler Opening line. The line comprised a feed table, coarse fibre opener, volumetric feeder, fine opener and lap former.

The lap once formed was fed into a worsted type carding machine - a Thibeu CA6 comprising a weigh pan hopper, fibre opening section and a main carding cylinder. The web of fibres formed was condensed into the form of a sliver with an average weight of 5 grammes per metre length.

The slivers were then attenuated on a conventional short-

5 staple draw frame - a Platts Globe Draw Frame - in which  
rollers operated at differential surface speeds to  
attenuate the multiple feed of slivers (6-8) into a uniform  
single sliver of uniform weight and thickness  
(approximately 3g per metre length).

10

The drawn sliver was converted into roving on a roving  
frame which further attenuates the sliver. Twist was  
inserted to add cohesion to the strand. The roving was  
then spun on a ring spinning machine in which further  
15 drafting took place and twist was inserted to form the  
final yarn.

5

## CLAIMS

1. An absorbent, composite fibre comprising a matrix of from at least 10% to less than 50% by weight of water insoluble alginate having dispersed therein at least 40% by weight of another polysaccharide.

2. An absorbent composite fibre as claimed in claim 1 wherein the water insoluble alginate is calcium alginate.

3. An absorbent composite fibre as claimed in claim 1 or claim 2 wherein the other polysaccharide is selected from the group comprising carboxymethyl cellulose, carboxyethyl cellulose, other derivatives of cellulose, cellulose, pectin, hyaluronic acid and chitosan.

20

4. An absorbent composite fibre as claimed any preceding claim wherein the fibres comprise from 40% to 90% by weight of another polysaccharide, preferably 60% to 85% by weight.

5. An absorbent composite fibre as claimed in any preceding claim wherein the fibres comprise from 70% to 80% by weight of another polysaccharide.

6. An absorbent, composite fibre as claimed in any preceding claim wherein the other polysaccharide is carboxymethylcellulose or pectin or a mixture thereof.

7. An absorbent, composite fibre as claimed in any preceding claim wherein the fibres comprise from 10% to 49%

5 by weight of the fibre of calcium alginate, preferably 30% to 40%.

8. An absorbent, composite fibre as claimed in any preceding claim wherein the fibres comprise from 20% to 30%  
10 by weight of the fibre of calcium alginate.

9. A method for making a composite, absorbent fibre comprising the following steps:

- (i) adding sodium alginate and another polysaccharide to  
15 water to form a dope;  
(ii) forcing the dope through a spinneret to form fibres;  
(iii) treating the resulting fibres with a source of ions to convert the alginate to water insoluble alginate and cross-link the alginate to the other polysaccharide;  
20 (iv) drying the fibres.

10. A method as claimed in claim 8 wherein the source of ions in step (iii) is calcium ions.

25 11. A wound dressing comprising an absorbent, composite fibre comprising a matrix of from 10% to less than 50% of water insoluble alginate having dispersed therein at least 40% of another polysaccharide.

30 12. A wound dressing comprising a mixture of discrete textile fibres and discrete absorbent, composite fibres said absorbent, composite fibres comprising a matrix of from 10% to less than 50% of water insoluble alginate having dispersed therein at least 40% of another

5 polysaccharide.

13. A wound dressing as claimed in claim 12 comprising  
from 50% by weight to 95% by weight of textile fibres and  
5% by weight to 50% by weight of absorbent, composite  
10 fibres.

14. A wound dressing as claimed in any claim 11 or claim  
12 comprising from 75% to 90% by weight of textile fibres  
and 10% to 25% by weight of absorbent, composite fibres.

15  
15. A wound dressing as claimed in claims 11 to 14 wherein  
the fibres are in the form of a woven fabric.

16. A wound dressing as claimed in claims 11 to 15 wherein  
20 the fibres are in the form of a carded web.

17. Use of a wound dressing as claimed in claims 11 to 16  
for the treatment of a wound by placing the dressing in  
direct contact with the wound.

25  
18. A wound dressing as claimed in claims 12 to 15 wherein  
the dressing is in the form of a knitted fabric comprising  
support yarn and in-laid yarn, the support yarn being  
substantially free of composite fibres.

30  
19. A wound dressing as claimed in claim 18 wherein the  
fabric is a warp knitted fabric.



# INTERNATIONAL SEARCH REPORT

Intern. Appl. No.  
PCT/EP 97/01880

<b>A. CLASSIFICATION OF SUBJECT MATTER</b> IPC 6    D01F9/04    D01F2/28    A61L15/28		
According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) IPC 6    D01F    A61L		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used)		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 96 10106 A (INNOVATIVE TECH LTD ;QIN YIMIN (GB); GILDING KEITH DENNIS (GB)) 4 April 1996 cited in the application see the whole document ---	1-19
A	DATABASE WPI Section Ch, Week 8814 Derwent Publications Ltd., London, GB; Class A32, AN 88-093952 XP002037006 & JP 63 042 917 A (AGENCY OF IND SCI & TECHNOLOGY) , 24 February 1988 see abstract --- <div style="text-align: right;">-/--</div>	1-19
<div style="display: flex; justify-content: space-between;"> <span><input checked="" type="checkbox"/> Further documents are listed in the continuation of box C.</span> <span><input checked="" type="checkbox"/> Patent family members are listed in annex.</span> </div>		
<div style="display: flex;"> <div style="flex: 1;"> <p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="flex: 1;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&amp;" document member of the same patent family</p> </div> </div>		
Date of the actual completion of the international search  <div style="text-align: center;">6 August 1997</div>	Date of mailing of the international search report  <div style="text-align: center;">19.08.97</div>	
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax (+ 31-70) 340-3016	Authorized officer  <div style="text-align: center;">Tarrida Torrell, J</div>	

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

Internat. Application No

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